Process for solids suspension and dosing in a high-pressure process

[0001] The invention relates to a process for solids suspension and dosing in a high-pressure process, for example colour pigments in such a high-pressure process using a supercritical fluid as process fluid at a pressure of >150 bar. The solids suspension step takes place at low pressure in a completely separate suspension process. The solids undergo a suspension or partial dissolution in a non-critical and liquefied gas. Said suspension pressure is as low as <90 % of the critical pressure of the liquefied gas. The dosing of the solids suspension in the high-pressure process takes place by means of a pump.

[0002] Various state-of-the-art techniques and devices are known what solids can be feed during a high-pressure process. It is common practice to feed the solids by means of a feed hopper, feed tank or similar device, said devices being flooded with the process fluid so that the solids are entrained to enter the high-pressure process.

[0003] DE 199 28 405 describes a process for dyeing a textile substrate in the presence of at least one supercritical fluid, including a dosing process in which the required quantity of powdered dyeing agent is divided into a multitude of small batches, each batch coming into contact with the supercritical fluid for that time necessary for solids dissolution or dispersion. The device described in the published document provides for an oscillating piston with a bearing on either side, the piston being placed across the stream and moved along the process line that contains the critical fluid. The said piston takes up a batch of solids through the first bore in the end position and in the opposite end position. This first bore is located in the main stream so that the solids are removed from the bore by the said fluid. At the other piston end, a second bore is simultaneously filled or emptied, respectively.

[0004] The process and device according to DE 199 28 405 have the disadvantage that the device must be rated and sized in accordance with the main process. Furthermore, the bore emptied and located in the main stream is used to catch supercritical gas and moved into the filling position so that this section must either have an equal or higher pressure or a flashing cycle must each time be performed which would inevitably impair the filling process. Moreover it is in fact critical to cross a high-pressure gasket with the bores guided by the piston. After short operating periods there will surely be damage to the gasket material, i.e. leakage problems will occur.

[0005] WO 97/13915 describes a very common method of dosing solids in a supercritical process. This process provides for a dye preparation vessel arranged in a by-pass of the main process stream. For removal of the solids the respective valves are opened and the solids are entrained by a part stream of the main process fluid. Hence, it is not possible that uniform dosing or fine tuning of the dying agent feed be carried out since the mixture of solids and fluid is diluted by the process fluid taken in. Moreover, all components arranged in this process by-pass must satisfy the pressure and temperature requirements of the main process.

[0006] A comparison with the suspension process optimised in accordance with WO 97/13915 is described in patent US 6,261,326. In said process a dye preparation vessel is used for the dissolution or suspension of the dying agent, with the aid of an agitator or a pump to circulate a partial quantity of the solids. As stated in the said document, there is an almost critical fluid state in this preparation process because the latter is fed with supercritical fluid from the main process. As in the case of the processes mentioned above, this preparation method also has a deficiency, i.e. it must meet the high-pressure requirements of the main process, which in this particular case is called treatment process. Moreover, solids dosing and input not shown in US 6,261,326 must be effected by a sophisticated hopper system or the solids feed vessel requires that the high pressure of the main process be met.

[0007] The aim of the invention, therefore, is to provide a solids suspension and dosing process that operates independently of the high-pressure cycle and with a higher degree of efficiency than that of the state-of-the-art processes.

[0008] The aim of the invention can be achieved by means of a suspension and dosing process for granulated or powdered solids to be fed to a high-pressure process which essentially uses a supercritical gas as process fluid. The said step constitutes a solids suspension stage of the high-pressure process and consists of a suspension tank and a device for liquid stirring, incl. the respective inlet and outlet lines.

[0009] When the process is implemented the first step provides for a suspension tank filled with solids and a fluid, the latter being a liquefied gas which is in a non-critical state.

[0010] In a second step, the solids become suspended in the fluid by means of a liquid agitating device and are kept in suspension. The liquid agitating device should be an agitator or a pump arranged on the outside of the suspension tank, said pump being connected to the vessel by an intake line and a delivery line and part of the suspension inventory being constantly circulated in a cycle.

[0011] The pressure in the suspension step should preferably be <90% of the critical pressure of the gas used, the ideal value being <60 bar so that a gaseous phase is present above the liquid phase, the ideal value being <60 bar so that a gaseous phase is present above the liquid phase.

[0012] The last step is required to pump the suspension into the high-pressure process. Hence, the pump and its delivery line connected to the main process must satisfy the high-pressure process requirements. Any process equipment mounted on the intake side of the pump merely needs comply with the lower standard.

[0013] An advantageous embodiment of the process according to the invention is to provide a fluid for the solids suspension which essentially is chemically identical with the process fluid of the high-pressure process.

[0014] To optimise the process further substances are added to the fluid. Said substances are, for example, cyclic and acyclic short-chain hydrocarbons or alcohols, aldehydes or ketones as well as H_2O and mixtures thereof.

[0015] For the process according to the invention it is recommended that the pressure applied in the suspension tank during the dosing cycle of the solids suspension into the high-pressure process be maintained at a preferably constant level by topping up with gas in the gaseous state.

[0016] An ideal process variant, therefore, permits the feed of the solids suspension into the high-pressure process to take place continuously during the vessel emptying cycle and, additionally, the concentration of the solids in the fluid to be maintained at a constant level or at a variable value with the aid of the pump delivery. The concentration in the suspension tank essentially remains constant during the emptying cycle as the top-up gas is not in the liquid phase but in the gaseous phase.

[0017] Another advantageous embodiment of the invention provides for solids that are soluable in the high-pressure process, such as colour pigments, bonding agents, bleaching agents, aromatic fluids or mixtures thereof. In this context the invention also bears an advantage to the extent that the solids - contrary to the start-of-the-art technology – is merely suspended and that the specific soluability of the respective feedstocks must not be met in the suspension stage. When the said feedstocks are added to the big volumetric streams of the main process, the said solids directly dissolve on account of the high concentrate rate.

[0018] Therefore, a beneficial embodiment of the invention is constituted by the fact that the suspension stream is adjusted at the admixture to the high-pressure process in such a manner that the ratio of the volumetric streams of suspension and high-pressure fluid is 1 : 50 and in the ideal version it is $\leq$ 1 : 100.

[0019] The suspension stream which is very small compared to the main-process volumetric stream, as a rule, has a negligible physical influence only on the main process.

[0020] The process according to the invention is illustrated on the basis of the two figures showing a typical process layout.

[0021] Figure 1 shows the suspension process using a pump to stir the liquid and Figure 2 depicts the process using an agitator without external cycle.

[0022] According to Figure 1, suspension process (1) connected to the high-pressure process (4) via line (6) essentially consists of suspension tank (2) and pump (3). Suspension tank (2) is filled with liquefied gas via line (7) and with solids via line (8). The layout in Figure 1 shows that liquid inventory (2a) and gas inventory (2b) form in suspension tank (2). A part stream is withdrawn from the tank by means of suspension pump (9) via line (10) and recycled via line (11).

[0023] When suspension tank (2) is emptied the solids suspension is conveyed via line (5) by means of pump (3) and via line (6) to high-pressure process (4). It becomes obvious that merely pump (3) and the delivery side of the pump, i.e. line (6), must be rated for the pressures applied in high-pressure process (4). The remaining components of the suspension process must merely comply with the requirements for pressures up to about 60 bar.

[0024] Figure 2 shows a further embodiment of the process, suspension tank (2) being equipped with agitator (13) so that a suspension can be prepared and kept stable.